Embrittlement Test Development
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Hydrogen Embrittlement Design of Experiments

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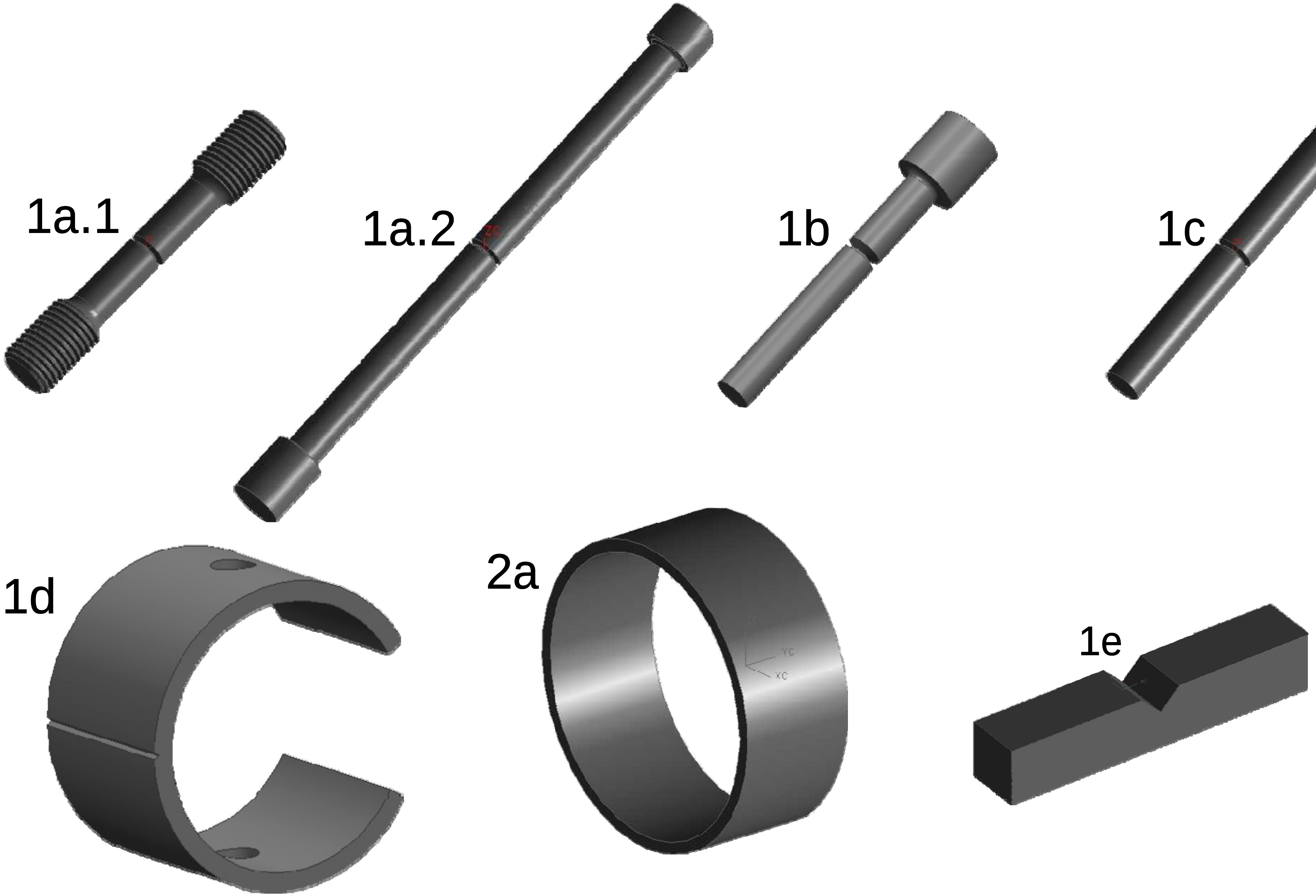
Hydrogen Re-Embrittlement Testing Issues

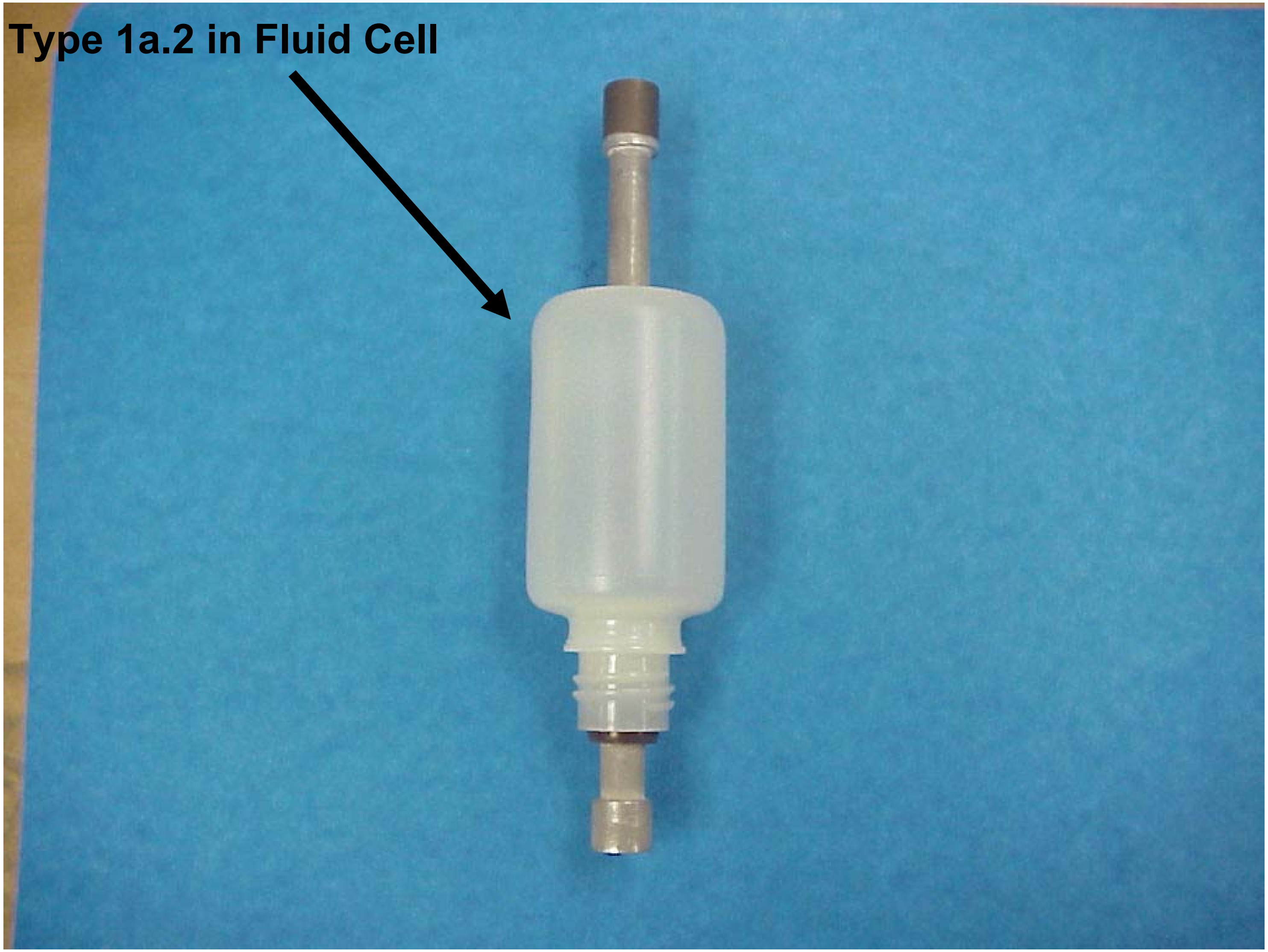
Background Info

DoD Metal Finishing Workshop May 2006 – Washington D.C.

- Workshop Identified Barriers for Implementing Cadmium Plating Alternatives
- Hydrogen Embrittlement Testing Was Identified as a Major Barrier
  - Need to Verify that Alternative Coating Process is Non-Embrittling to HSS (Called Embrittlement Testing)
  - Need to Verify that Maintenance Chemicals on HSS with Alternative Coating Do Not Embrittle the HSS (Called Re-Embrittlement Testing)
- Chuck Pellerin Agreed to Provide “Seed Money” to Solve Problem

ASTM F 519 Specimens




Type 1a.2 in Fluid Cell

Re-Embrittlement Test Issues

- Not Standardized Across the Industry – **Variations of ASTM Annex A5 Used**
  - Various ASTM F519 Specimens Used
    - Type 1.a.1, 1.a.2, 1b, 1c, 1d, 1e, and 2a
  - Various Specimen Immersion Methods
    - Wet for 150 hrs, Wet Than Dry, Concentrated or Diluted Chemicals
    - Volume of Fluid, Temperature
  - Various Loading Methods
    - Tension, Bending, Sustained Load, Incremental Step Loading (24 hours)
    - 45%,65%,75% NFS, 80%YS for 150 or 200 hrs
  - Various Strength Levels to Bake or No-Bake
    - 160 ksi, 180 ksi, 200 ksi, 220 ksi

Re-Embrittlement Testing Is Our Tower of Babel

The Confusion of Tongues by Gustave Doré (1865)

Project Team

- Project Funding from SERDP
- ASTM-F07 committee on Aerospace and Aircraft, Subcommittee 04 on HE Members governing specification ASTM-F-519 on Hydrogen Embrittlement testing
- Scott Grendahl, US Army Research Laboratory - Project Lead (Overhead Only)
- Ed Babcock, Boeing-Mesa - Technical assistance (UNF)
- Steven Gaydos, Boeing-St. Louis - Technical assistance (UNF)
- Joe Osborne, Boeing-Seattle - Technical assistance (UNF)
- Lance Weihmuller, Bell Helicopters - Technical assistance (UNF)
- Stephen Jones, Boeing Seattle - Data analysis (Boeing Internal Funding)
- Green Specialty Service Inc. - Specimen fabrication
- Omega Research Inc. - Test performer
- ASKO Plating Company – Specimen Plating

Green Specialty Service, Inc.

Omega Research Inc.

Bell Helicopter A Textron Company

SERDP

Technical Objective

- **Phase I – Eliminate ASTM-F-519 hydrogen embrittlement testing ambiguities via the generation of comparative data sets across multiple geometries**
- **Utilize data sets to consolidate test geometries and provide rationale for refining ambiguous test procedures**
- **Phase II and III assess specific maintenance chemicals in terms of concentration, hydrogen bake relief dwell, times and temperatures, other materials (300M, Aermet 100), and coatings**
  - **Widely known and accepted in the aerospace community that test procedures and resulting data vary based on the "grey areas" of the specification and geometry used**
    - **Materials – air melt vs. aerospace grade 4340**
      - **Air melt not really available**
    - **Specimen load levels not equivalent**
    - **Specimen notch K_I not equivalent**
    - **519 might be too stringent for lower strength applications**
  - **Phase II and III will mitigate restrictions on widely used chemicals and coatings, as well as increase future testing uniformity through spec changes which will lower the existing perceived risk in high strength components**

Technical Approach

- Team devised a "Design of Experiments, DoE" approach since matrix was large and funding was minimal
- Study focused on 3 variables for 5 geometries
  - Material strength level (ksi) – (Hardness) Range 140 – 280 ksi
  - Test load (% Notch fracture strength, NFS) Range 40 - 80% NFS
  - Hydrogen solution concentration (wt% NaCl) 1.27E-5 - 3.5 wt% NaCl
    - 0% not used as low end since solutions lacking ions were proven very aggressive

Condition	-α	-	0	+	+α
Strength (ksi)	140	158	210	262	280
Test Load (%NFS)	40	45	60	75	80
NaCl Conc (wt% NaCl)	1.27E-05	0.01	0.50	2.36	3.50

- Requires 81 specimens per geometry instead of 125 and generates predictive model for entire range and beyond.

Technical Approach

- DoE in 3 Steps
  - Estimate first order model, test if second order model is required
  - Add test runs to estimate second order model
  - Add confirmation runs to see how well prediction works

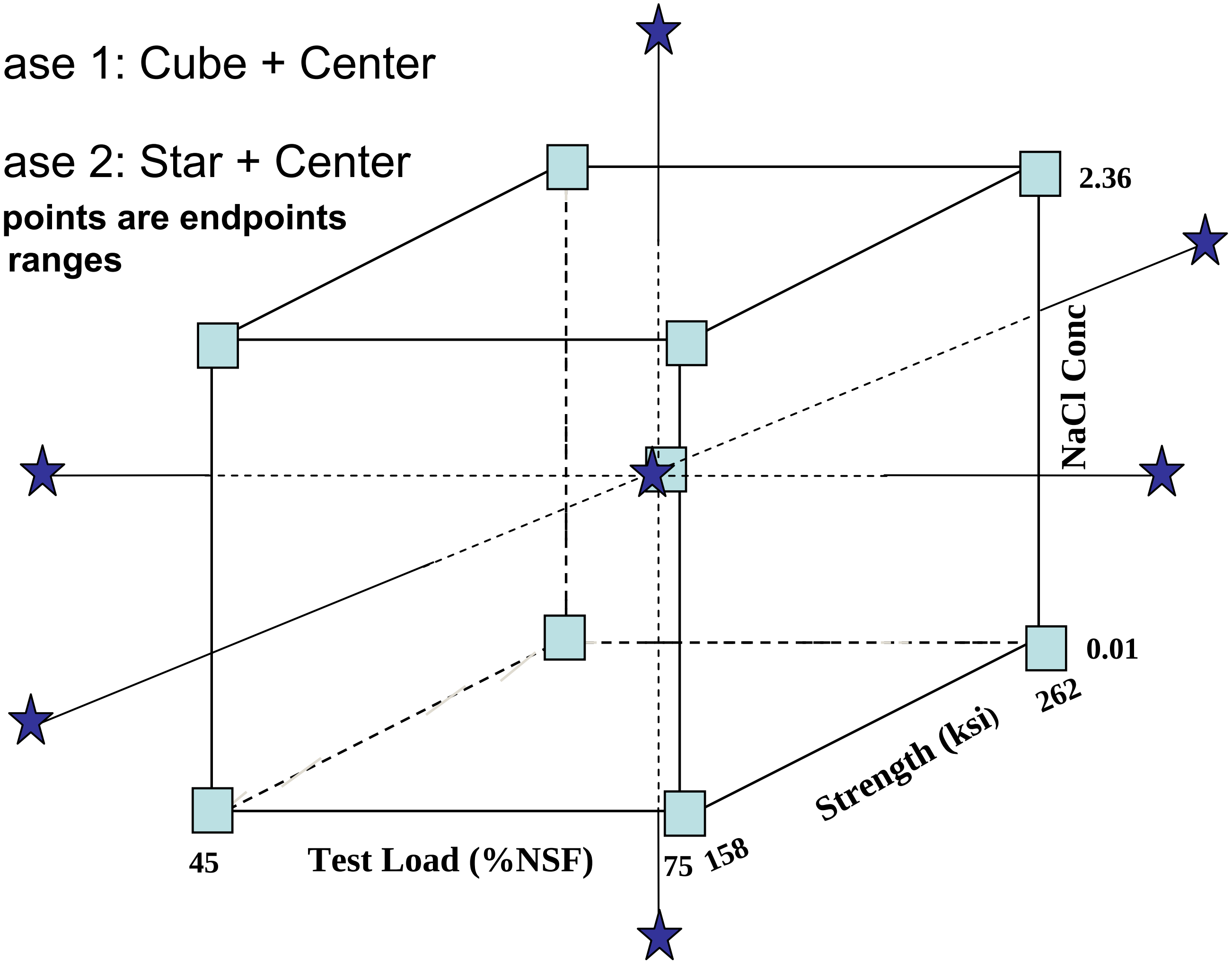
- Analysis combines data from all three steps
  - Step 1 - Full factorial in two levels, repeated twice, with 12 center points
  - Step 2 - Star points for each factor, repeated 5 times, with 2 center points
  - Step 3 - 18 additional tests for confirmation

Experimental Design

Phase 1: Cube + Center

Phase 2: Star + Center

- Star points are endpoints of the ranges



Linear Portion Test Matrix					
		A	B	C	Run Order
Repeat entire matrix 2x for 1a.1, 1a.2, 1c, 1d and 1e	RUN ID	Strength (ksi)	Test Load (%NFS)	NaCl Conc (wt% NaCl)	
Linear Portion	L1	-	-	-	Random
	L2	-	-	+	
	L3	-	+	-	
	L4	-	+	+	
	L5	+	-	-	
	L6	+	-	+	
	L7	+	+	-	
	L8	+	+	+	
Center Points	C1	0	0	0	
	C2	0	0	0	
	C3	0	0	0	
	C4	0	0	0	
	C5	0	0	0	
	C6	0	0	0	

Quadratic Portion Test Matrix					
		A	B	C	Run Order
Repeat Q1-Q6 5x for 1a.1, 1a.2, 1c, 1d and 1e	RUN ID	Strength (ksi)	Test Load (%NFS)	NaCl Conc (wt% NaCl)	
No Repeats	C7	0	0	0	First
Quadratic Portion	Q1	+α	0	0	Random
	Q2	-α	0	0	
	Q3	0	+α	0	
	Q4	0	-α	0	
	Q5	0	0	+α	
	Q6	0	0	-α	
No Repeats	C8	0	0	0	Last

		A	B	C	
	RUN ID	Strength (ksi)	Test Load (%NFS)	NaCl Conc (wt% NaCl)	Run Order
Confirm Portion	1	T5	44	0.5	Random
	2	T4	80	3.55	
	3	T4	74	2.36	
	4	T4	71	0.50	
	5	T4	71	0.01	
	6	T4	65	0.01	
	7	T4	57	0.01	
	8	T4	51	0.01	
	9	T3	80	3.5	
	10	T3	80	0.5	
	11	T3	71	2.36	
	12	T2	90	3.5	
	13	T2	87	2.36	
	14	T2	88	0.5	
	15	T2	81	3.5	
	16	T2	90	0.01	
	17	T2	90	0	
	18	T2	80	2.36	

Prior and Leveraged Work

- Boeing Ruggedness Study
  - Aimed at establishing which factors were most important
    - Surface condition plated or bare
    - Notch condition plated or bare
    - Solution Volume
    - Solution Temperature
    - Solution Concentration
    - Exposure Time
    - Exposure Temperature
- Boeing Risk Reduction Study
  - 1a1 and 1d geometries at 519 strength and load levels
  - Assessment of NaCl solution merit, low strength material procedure
- SPOTA/ARL for re-machining and Aerospace Grade material purchase
- ASTM Committee and coordination work - unfunded
- ASKO Plating for developmental work
- Boeing and ARL labor to date

Results

- 1a2 Testing
  - Low strength material did not fail
  - Higher strength materials had most failures, as expected
  - Data for Steps 1+2 were combined for analysis and prediction
  - Step 3 - Confirmation tests were run where predicted failure times were within 168 hours
  - Same NaCl levels were utilized, although could have been varied

- 1d Testing on-going

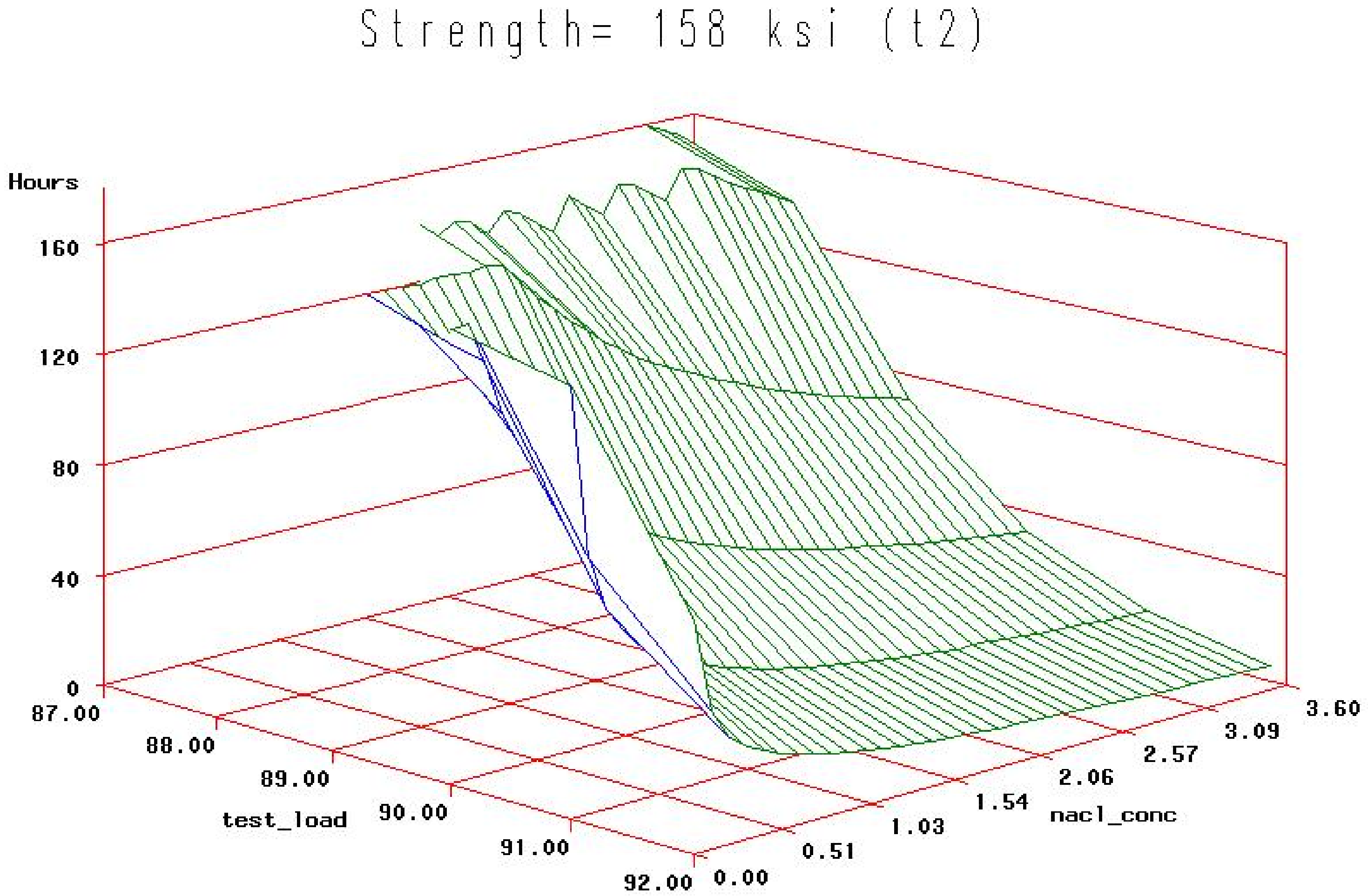
- 1a1, 1c, and 1e will be completed when machining is finished

- Model for 1a2 reflecting completed Steps 1-2
  - Y=ln X = 9.11+(-6.70*strength)+(-5.61*test_load)+(-0.16*NaCl_conc) + (2.10*strength*test_load)+(-1.21* test_load*NaCl_conc) + error

- Model adjusted to reflect Step 3 confirmation runs for greater accuracy
  - Y=ln X =11.15 -11.81*strength -7.02*test_load -0. 80*NaCl_conc +3.46*strength*test_load +2.84*strength ^2 + error

Results

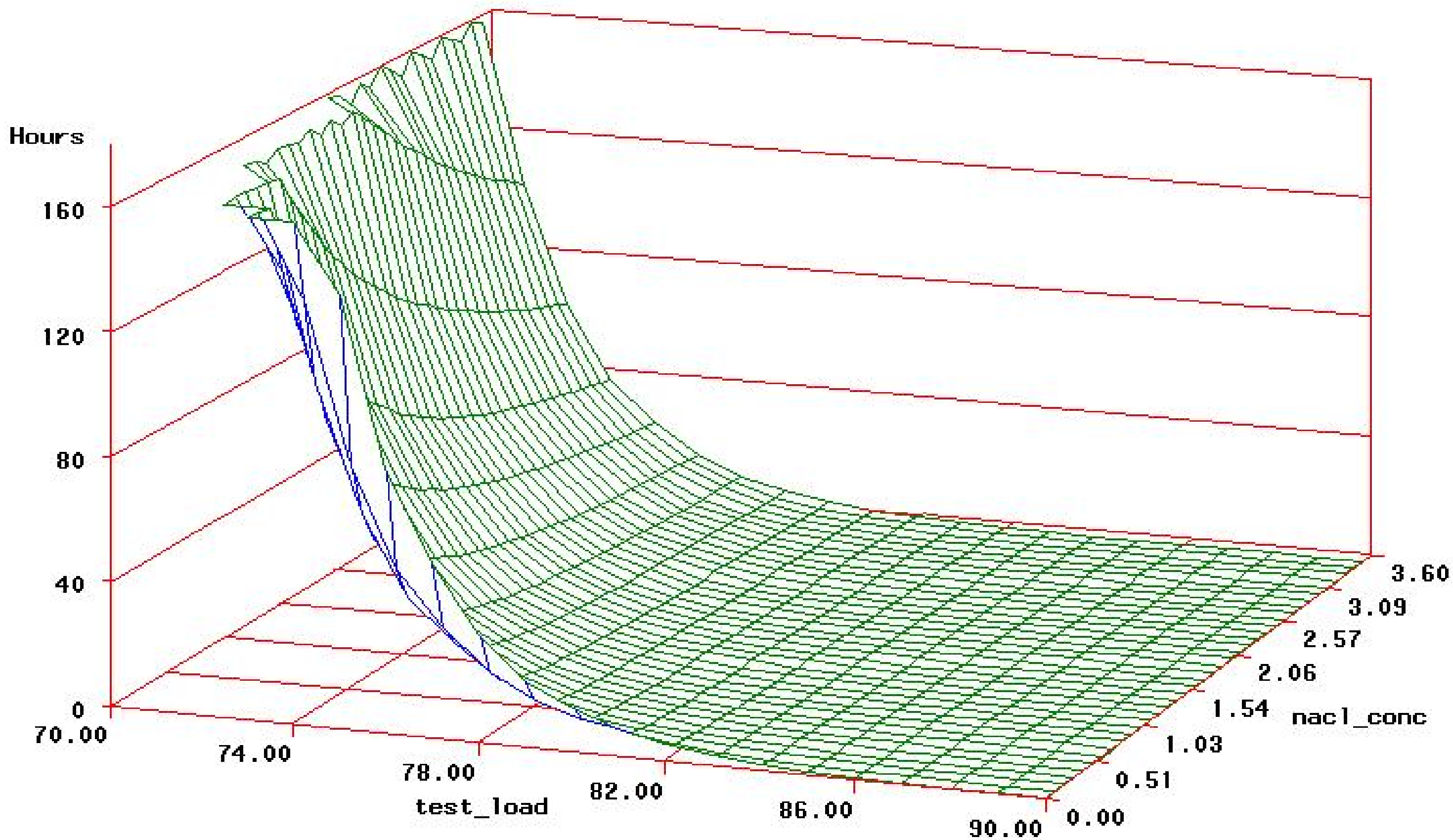
Predicted Median Lifetime



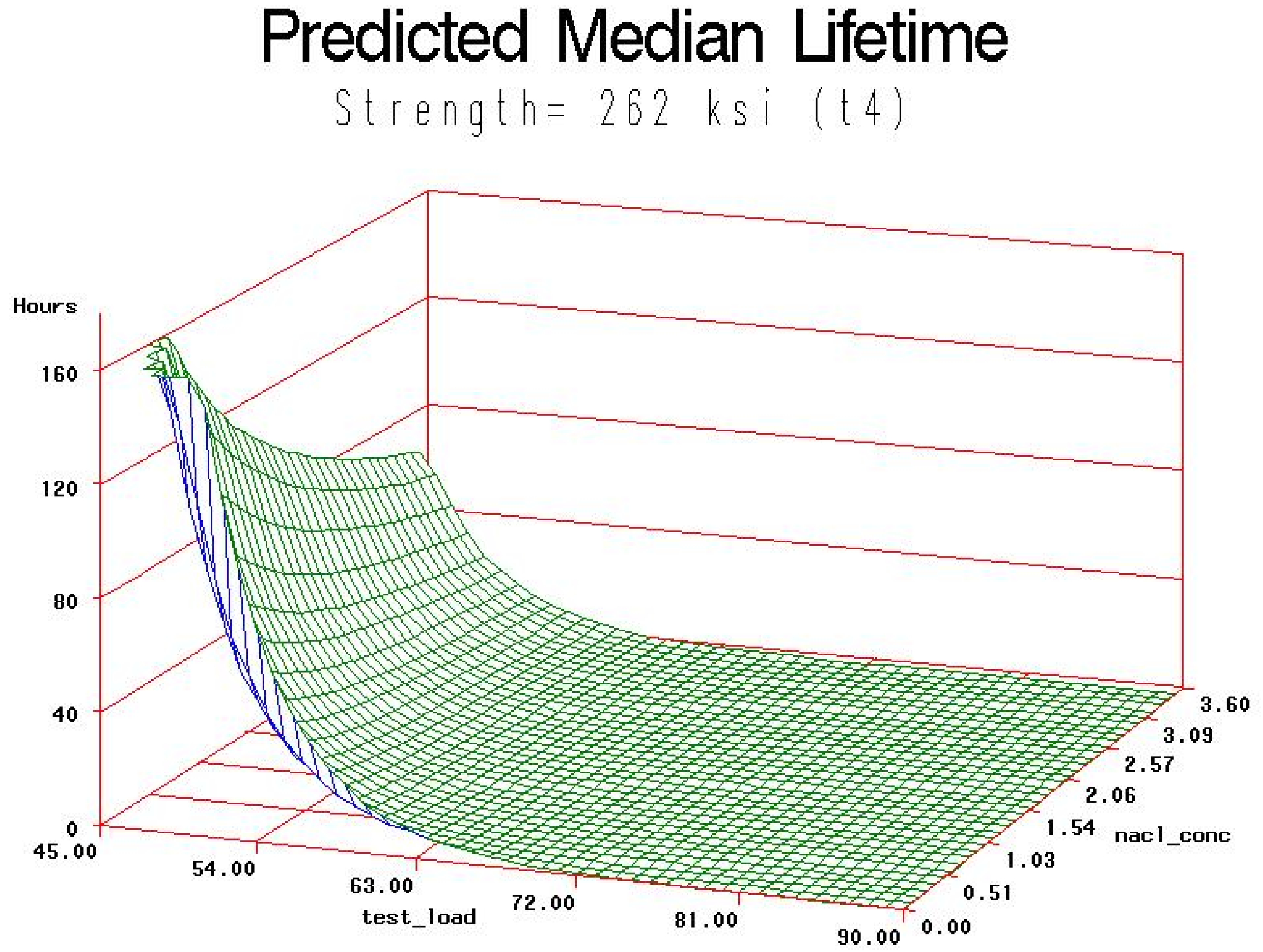
Results

Predicted Median Lifetime

Strength= 210 ksi (t3)

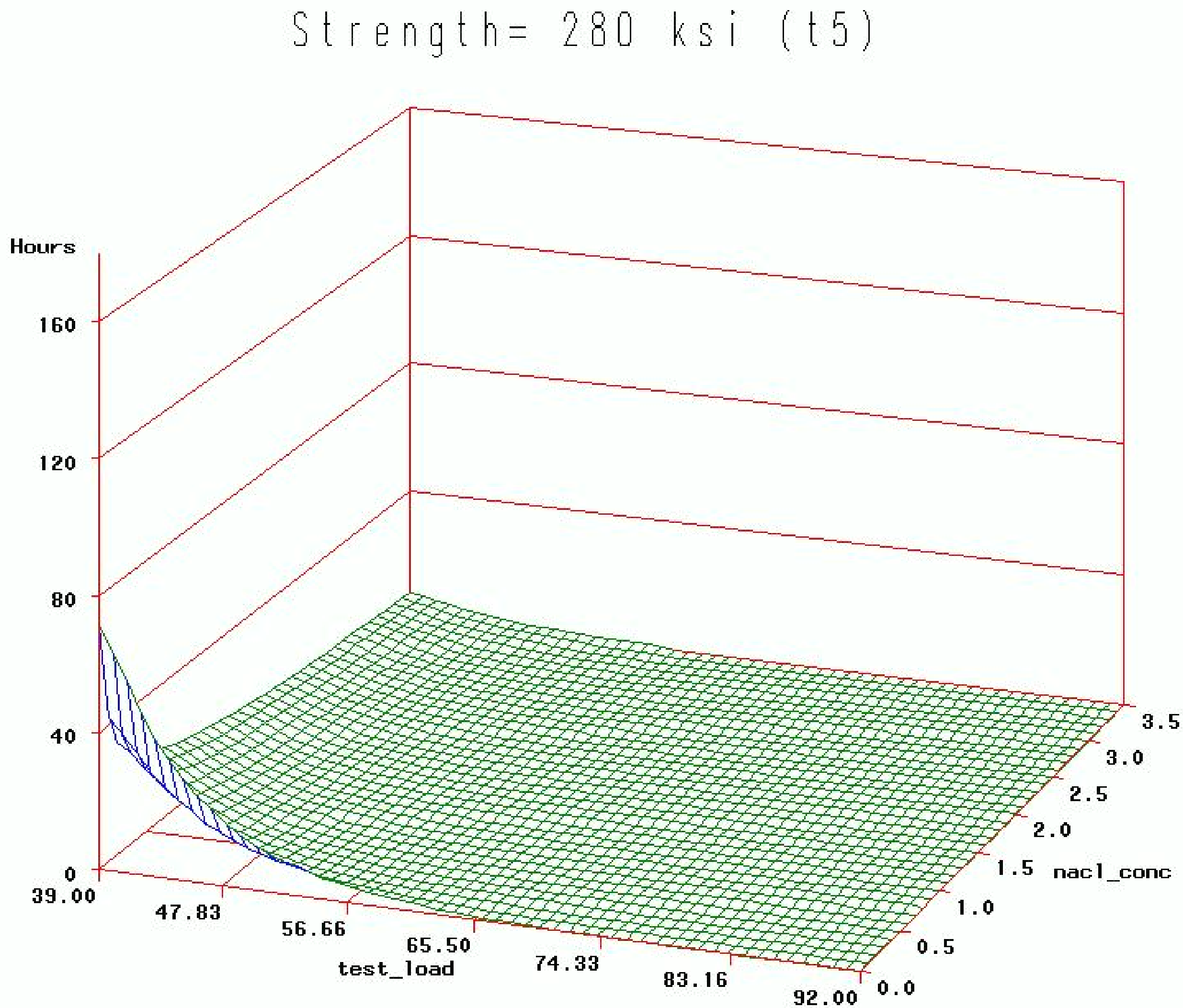


Results


Predicted Median Lifetime
Strength= 262 ksi (t4)
Hours
160
120
80
40
0
45.00
54.00
63.00
72.00
81.00
90.00
test_load
0.00
0.51
1.03
1.54
2.06
2.57
3.09
3.60
nacl_conc

Results

Predicted Median Lifetime



Results - Confirmation Runs

Strength (ksi)	Test Load (%NFS)	NaCl Conc (wt% NaCl)	Specimen S/N	Predicted TTF (hrs)	Actual TTF (hrs)	NFS	Hours	Comments
T5	44	0.5	5	16	0.6166667			low test load, but high strength
T4	80	3.55	9	0	0.05			Great
T4	74	2.36	3	0	0.0833333			Great
T4	71	0.50	14	1	0.15			Great
T4	71	0.01	13	2	1.4			Great
T4	65	0.01	2	5	4.5			Great
T4	57	0.01	11	14	74.9			Specimen anomaly?
T4	51	0.01	4	36	NF	66%	1.8	low test load
T3	80	3.5	10	0	1.6			Great
T3	80	0.5	20	4	3.4			Great
T3	71	2.36	36	45	27.4			Great
T2	90	3.5	14	0	46.5			NaCl plays less role
T2	87	2.36	9	0	NF	97%	1.95	NaCl plays less role
T2	88	0.5	5	2	NF	88%		Low matl strength
T2	81	3.5	7	9	NF	86%	2	NaCl plays less role
T2	90	0.01	11	13	NF	95%	0.18333	
T2	90	0	10	32	0.0083333			test load is MOST improtant
T2	80	2.36	15	40	NF	85%	2	NaCl plays less role

Yellow - At least we did get failures. Our original model just needs adjustment for influence.
- Model will improve with time and more data points.

Results

- **Test load and material strength level are the most influential factors**
- **Agrees with previous Risk Reduction work**
- **NaCl solution (hydrogen generation) plays less of a role**
- 1d data plots will be generated from testing on-going
- Full matrix will be tested 1a1, 1a2, 1c, 1d, and 1e once identical heat treatment specimens are fabricated.
  - Approximately 50% completed
  - Presently at Heat Treated blanks stage

Transition Plan

- **Proved that a functional model for time to failure can be created**
  - Once models for all geometries at identical HTs are completed, consolidation/ambiguity reduction within the specification will be performed through ASTM F07.04 committee
  - Will remove inconsistent procedures/results and the inherent risk that arises
- **Matrix will be repeated with Aerospace Grade material**
  - Actual practice reflected
- **Procedure will then be in place to address the prospective, coatings, maintenance chemicals, and alternate materials/strength levels**
  - The additional data will allow the current restrictions for the use of the coatings and chemicals in the field, depots, and even at OEMs to be mitigated through the AMCOM/AMRDEC approval chain

Transition Plan

- Prospective coatings, maintenance chemicals, bake relief times and temperatures, and alternate materials/strength levels
  - Currently being restricted or limited by the requirement of post use bake relief due to lack of data
  - ASTM F 519 does not address concentration factors, material strength levels, bake relief
    - Only a worst case (pass/fail) approach is evaluated
    - This limits use of alternates
  - AMCOM/AMRDEC willing to review data and revise restrictions
  - Easing of the restrictions in field, depots, and OEMs will increase use
- Approval process governs Army Aerospace community but benefits everyone

Issues

- Phase I - Heat treating was improperly performed by a second source vendor. Only two geometries could be salvaged, 1a2 and 1d

- This caused a delay of 9 months to re-machine specimens

- Additional cost was mitigated with SPOTA/ARL funding for the Aerospace Grade 4340. Since additional specimens were being made, costs were minimized

- These two runs 1a2 and 1d, allow us to verify that the DoE will work, without having the expense of the entire matrix

- Since we must compare all geometries with exact heat treatment and cadmium plating, matrix is being entirely repeated

BACKUP MATERIAL

These charts are required, but will only be briefed if questions arise.

Acronyms and Symbols

- ARL – US Army Research Laboratory, APG, MD
- 1a1, 1a2, 1c, 1d, 1e
  - ASTM-F-519 test specimen geometries
- HE - Hydrogen Embrittlement
- AG – Aerospace Grade 4340 steel
- NFS - Notch Fracture Strength
- NaCl - Sodium Chloride
- AMCOM - US Aviation and Missile Command, Redstone Arsenal
- AMRDEC - US Aviation and Missile Research Development and Engineering Center, Redstone Arsenal

Publications

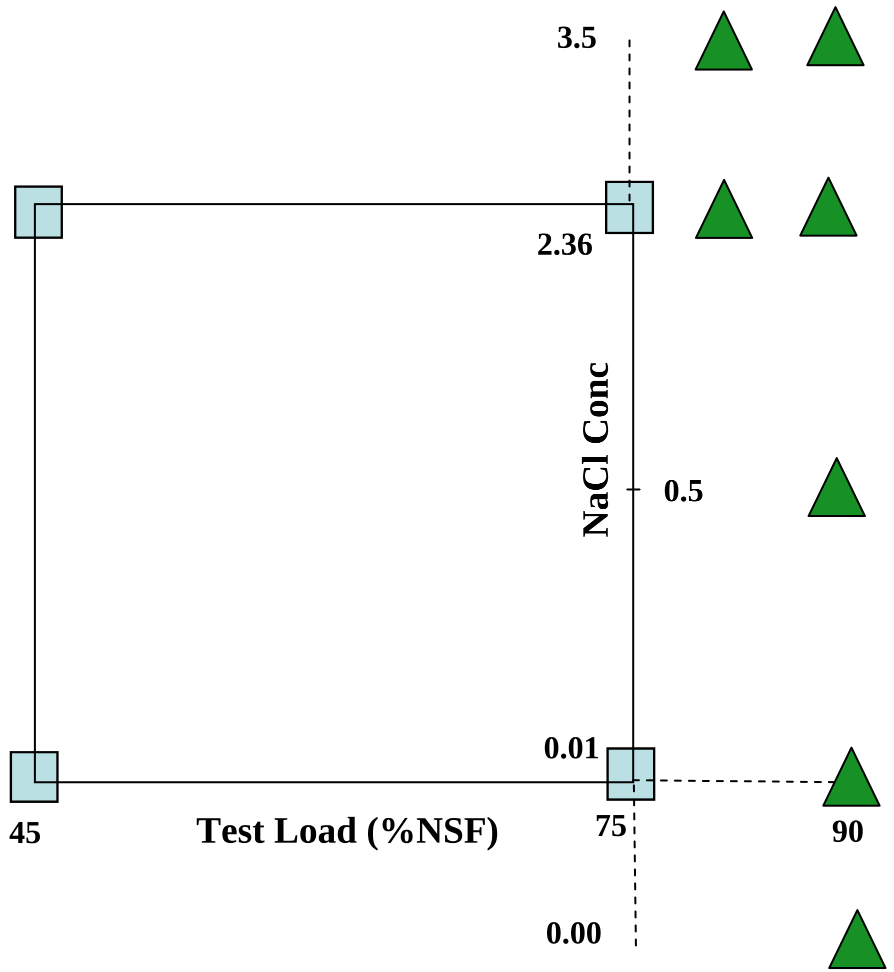
- Barron, J., “Effect of Coatings on the Structural Integrity of Fasteners”, ASTM F16.96 workshop, Northrop Grumman Shipbuilding, Newport News, 20 May 2009.
- Gaydos, S., “ASTM F 519 Annex A5 DoE Test Plan Status”, Presentation at ASTM F07.04 Subcommittee Meeting, The Boeing Company, St. Louis, MO, 15 April 2008.
- Babcock, E. A., “Aqueous Cleaning of High Strength Steel”, Whitepaper WP 3M11:07-051, To AED, The Boeing Company, Mesa, Arizona, 24 May 2007
- Gaydos, S., “SERDP Hydrogen Re-Embrittlement DoE Test Plan Status”, Presentation to DoD Metal Finishing Workshop – Chromate Alternatives for Metal Treatment and Sealing, The Boeing Company, St. Louis, 17 May 2007
- Babcock, E. A., “Update on ASTM F 519”, Presentation to AMCOM G-4 OEM at Redstone Arsenal, The Boeing Company Mesa, Arizona, April 24-25, 2007
- Babcock, E. A., “Annex 5 Ruggedness DoE Results + SERDP”, Presentation to ASTM International Committee F07.04 on Hydrogen Embrittlement, The Boeing Company, Mesa, Arizona, 17 April 2007
- Babcock, E. A., “Codifying Hydrogen Embrittlement Testing Protocols - Sound Tools for Alternatives Testing”, Presentation at Seventeenth Annual Cleaner Sustainable Industrial Materials & Process (CSIMP) Workshop, The Boeing Company, Mesa, Arizona January 21, 2007
- Babcock, E. A., “Hydrogen Embrittlement Testing and Evaluation: Progress and Status of Ongoing Research and Development”, Presentation to ASTM International Committee F07.04 on Hydrogen Embrittlement, The Boeing Company, Mesa, Arizona, 15 November 2006.

Results

Phase 1: Cube + Center

Confirmation Runs

T2

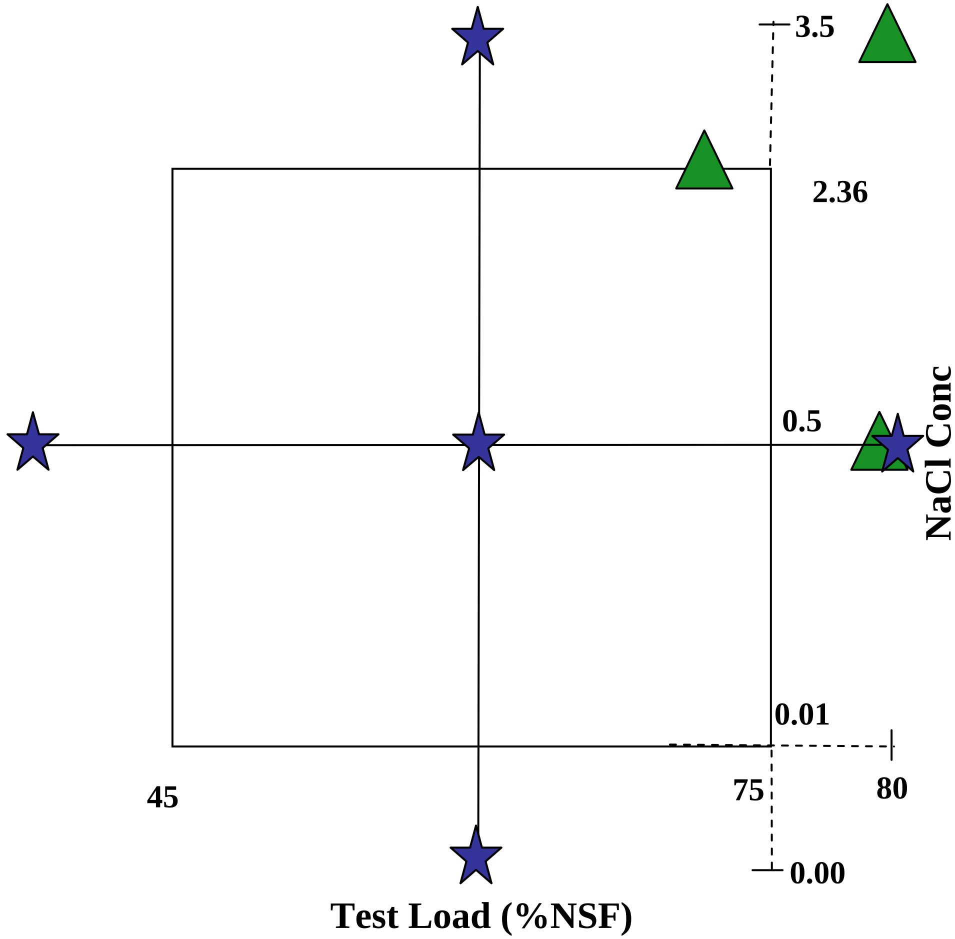


Results

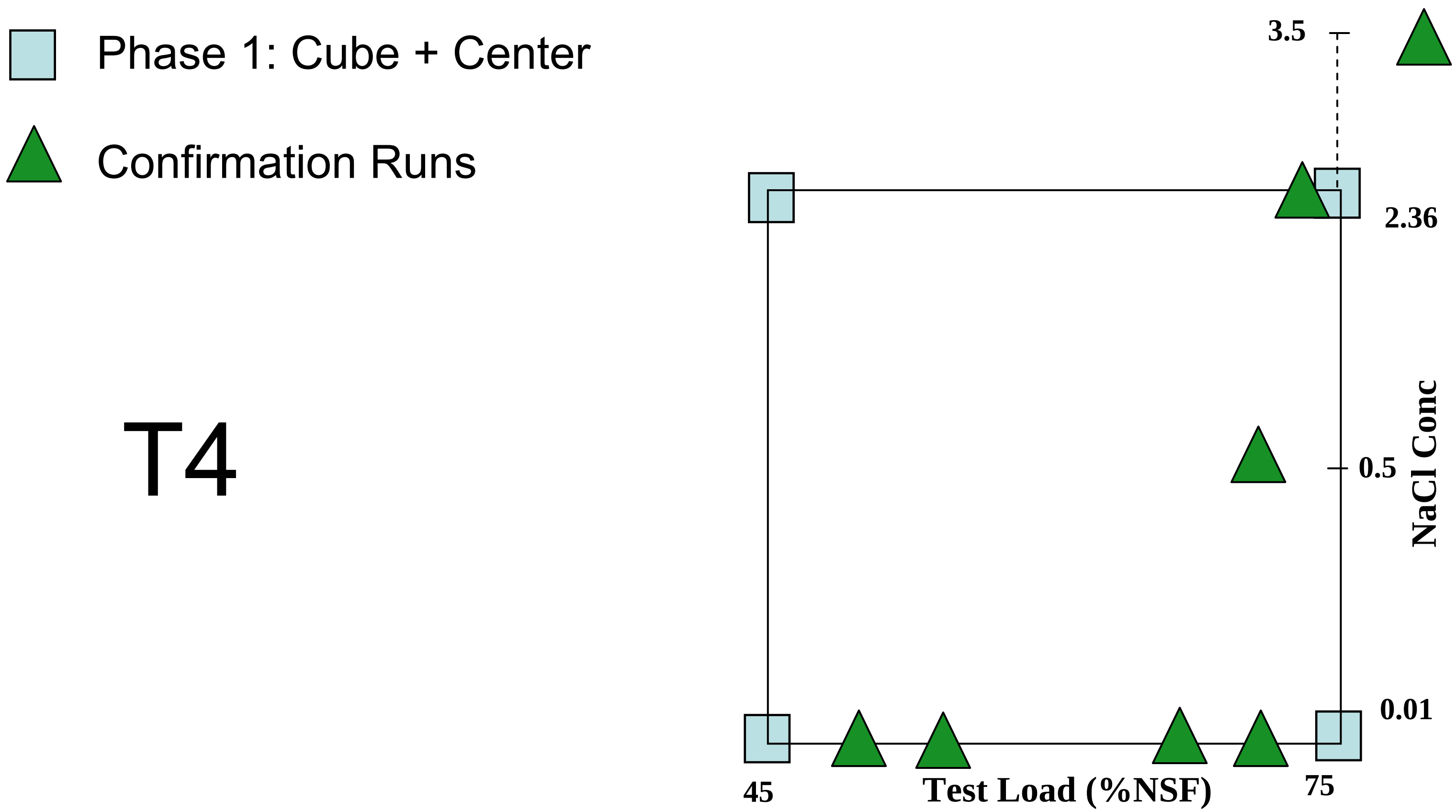
Phase 2: Star + Center

Confirmation Runs

T3



Results



Phase 1: Cube + Center
Confirmation Runs
T4
3.5
2.36
0.5
0.01
NaCl Conc
45
Test Load (%NSF)
75

Results

Phase 2: Star + Center

Confirmation Runs

T5